



TUC-26A/PCT/US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: James W. Dobson, Jr. et al.

Serial Number: 10/530,793

Group Art Unit: 1712

Filing Date: 04/08/2005

Examiner: TUCKER, PHILIP C.

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AFFIDAVIT UNDER 37 CFR 1.131

STATE OF TEXAS     )

COUNTY OF HARRIS   )

We, James W. Dobson, Jr., Kim O. Tresco, and Jorge M. Fernandez, employees of Texas United Chemical Company, LLC., being duly sworn, do hereby state that:

- (1) We are jointly co-inventors of the patent application identified above together with Bradley L. Todd and Trinidad Munoz, Jr., employees of Halliburton Energy Services, Inc.
- (2) Texas United Chemical Company, LLC. (hereinafter referred to as "TUCC") and Halliburton Energy Services, Inc. (hereinafter referred to as "HES") entered into an agreement to jointly develop an oil well drilling and servicing fluid (hereinafter referred to as "OWDSF") containing a magnesium oxide (magnesia) bridging agent.
- (3) Upon evaluating various samples of magnesia submitted by several companies, it was determined that the effective submitted magnesia had an Activity Index greater than 40 minutes.

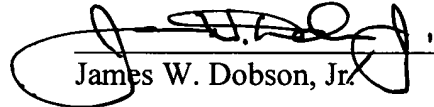
- (4) U.S. patent application SN 10/411,640 was filed on 04/11/2003 disclosing and claiming OWDSF containing a magnesia bridging agent having an Activity Index greater than 40 minutes.
- (5) The TUCC employees set forth herein determined that the Activity Index of the magnesia bridging agent decreased on reducing the particle size of the magnesia resulting in an Activity Index greater than about 800 seconds for an effective magnesia bridging agent. See lab notebook pages 47 and 59, book no. 165, attached (Attachment A).
- (6) (a) Roy F. House, agent for applicants, received a fax from James W. Dobson, Jr. on 07/14/2004 which indicated that the Activity Index of the magnesia bridging agent should be greater than 800 seconds (See Attachment B).
- (b) On 07/28/2004 applicants' agent sent a proposed patent application to the TUCC inventors and to the Patent Dept. of HES claiming a magnesia bridging agent having an Activity Index greater than 800 seconds.
- (c) International patent application was filed in the USPTO as PCT/US2004/033237 on 10/08/2004.
- (d) The present patent application identified herein was filed as a 371 of PCT/US2004/033237 on 04/08/2005.
- (7) (a) During the development of the magnesia bridging agent, TUCC's inventors identified hereinbefore independently discovered that citric acid

added to the OWDSF containing the magnesia bridging agent increased the low shear rate viscosity of the OWDSF.

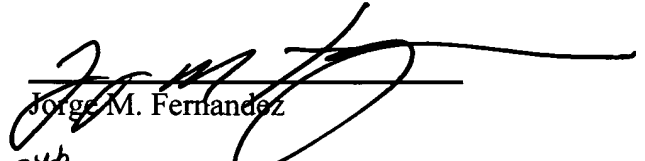
(b) A patent application had been prepared disclosing and claiming this invention (wherein the magnesia had an Activity Index greater than 40 minutes) when the discovery of the Activity Index of the effective magnesia bridging agent (i.e., >800 seconds) was made.

(c) The Activity Index of the magnesia bridging agent in the application was changed to greater than 800 seconds and the application was filed on 07/29/2004 under SN 10/901,822 (published patent application 20050003966).

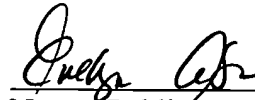
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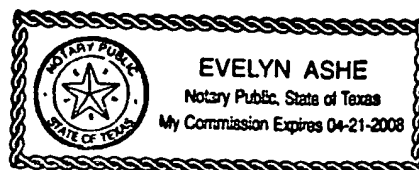
  
James W. Dobson, Jr.

  
Kim O. Tresco

  
Jorge M. Fernandez

Sworn to and subscribed before me this 19<sup>th</sup> day of FEBRUARY, 2007.

  
Notary Public  
My Commission expires: 4/21/08



From Page No. 46MAGNESIUM OXIDE  
CMA ACTIVITY TESTPrinciple

The rate at which magnesium oxide reacts with a dilute solution of acetic acid is used as a measure of activity. An excess of magnesia is used so that at the end point of the reaction, the solution goes from acidic to basic and is detected by a color change employing phenolphthalein indicator.

Apparatus and Reagents

Acetic acid solution 1.00 ± 0.01N, standardized \*  
Phenolphthalein soln. (1% solution in ethanol)  
Waring blender, 2 speed with 32 oz. glass container  
Balance with sensitivity of 0.01 gms.  
Stopwatch  
Thermometer  
Graduated cylinders, 100 ml and 500 ml

Procedure

1. Prior to the test, the water and the acetic acid solution should be brought to a temperature of  $25 \pm 1^\circ\text{C}$ .
2. Weigh a  $5.00 \pm 0.02$  grams aliquot of the magnesia sample.
3. Measure out 300 mls of water in a graduated cylinder and add it to the blender.
4. Carefully hold a thermometer in the blender and run blender until the temperature of the water is  $28^\circ\text{C}$ .  -  $82.4^\circ\text{F}$
5. Add 5-10 drops of phenolphthalein indicator solution.
6. Add the magnesia sample and immediately start the blender on low speed.
7. Count ten seconds from the start of the blender and add 100 mls of the 1.00N acetic acid solution. The stopwatch is started as the acid is being added.
8. Stop the timer when the solution turns to a definite pink color. Record the reaction time in seconds as the CMA activity.
9. Note: add three to five additional drops of indicator solution to the blender every 30 seconds until the color change has taken place.

\* available from Fisher Scientific, Catalogue # S0-A-36

98 HR 6 seconds  
5 mm P98 12:30-14:00 min  
10 mm P98 22:00-23:30 min  
20 mm P98 38:30-39:30 min

P98 ground  
@ plant

11.76 mm 22:30-23:30 min  
19.98 mm 58:00-59:30

- compare to procedure on page 38  
of this book

Scale 018

Therm 035

- also see book 161 pg 57

- also see book 148 pg 180

- also see book 165 pg 59

To Page No. 48

Witnessed &amp; Undersigned by me,

Date

Invented by

Date

Recorded by

04/28/04

From Page No. 57 47 Act Ind.

Principle

The rate at which magnesium oxide reacts with a dilute solution of acetic acid is used as a measure of activity. An excess of magnesia is used so that at the end point of the reaction, the solution goes from acidic to basic and is detected by a color change employing phenolphthalein indicator.

Apparatus and Reagents

- Acetic acid solution  $1.00 \pm 0.01N$ , standardized
- Phenolphthalein solution (1% solution in ethanol)
- Waring blender, 2 speed with 32 oz glass container
- Balance with sensitivity of 0.01 grams
- Stopwatch
- Thermometer
- Graduated Cylinders, 100 and 500 ml

Procedure

- Prior to the test, the water and the acetic acid solution should be brought to a temperature of  $25 \pm 1^\circ C$ .
- Weigh a  $5.00 (\pm 0.02)$  grams aliquot of the magnesia sample.
- Measure out 300 mls of tap water in a graduated cylinder and add it to the blender.
- Carefully hold a thermometer in the blender and run blender at medium low speed on the rheostat until the temperature of the water is  $28^\circ C$ .
- Add 5-10 drops of phenolphthalein indicator solution.
- Add the magnesia sample and immediately start the blender on low speed.
- Count ten seconds from the start of the blender and add 100 mls of the  $1.00N$  acetic acid solution. The stopwatch is started as the acid is being added.
- Stop the timer when the solution turns to a definite pink color. Record the reaction time in seconds as the CMA activity.
- Note: add three to five additional drops of indicator solution to the blender every 30 seconds until color change has taken place.

Magox Super Prem.  
tested on 12/22/04  
- 16 seconds.

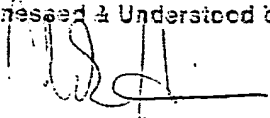
Magox P98 size	Old P98 w/1.0N Acetic Acid	Old P98 w/0.10N Acetic Acid	New P98 w/0.10N Acetic Acid
5µm P98	840 seconds	65 seconds	-
10µm P98	1,410 seconds	140 seconds	120 seconds
20µm P98 1740	2,370 seconds	200 seconds	200 seconds
30µm P98	1890 seconds	200 seconds	200 seconds

Conclusion:  
50µm P98 2,940 seconds  
150µm P98 5,610 seconds  
new - P98 ground plate =

scale 002  
therm 015

Even though P98 magnesium oxide is a dead-burned material, the reactivity is still different for different sized magnesium oxides. As the material gets finer, because of the increase in surface area, the reactivity of the material will increase. In the results above the 0.10N Acetic Acid is probably too weak an acid to differentiate well between particle sizes above 20µm.

also size  
pg 67  
To Page No. 62

Witnessed & Understood by me, 	Date 6-29-04	Invented by Ar M. T.	Date 6/25/04
		Recorded by	

**TBC-BRINADD**

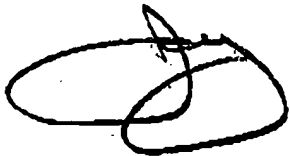
A DIVISION OF TEXAS UNITED CHEMICAL COMPANY, LLC

ATTACHMENT B**Fax**

To: ROY HOUSE From: JAY DOBSON  
Fax: \_\_\_\_\_ Pages: \_\_\_\_\_  
Phone: \_\_\_\_\_ Date: \_\_\_\_\_  
Re: \_\_\_\_\_ CC: \_\_\_\_\_

☐ Urgent☐ For Review☐ Please Comment☐ Please Reply☐ Please Recycle

ROY,  
APPLICATION FOR MAGNESIUM OXIDE BRIDGING  
PARTICLES SPECIFIES AN ACTIVITY INDEX  
OF GREATER THAN 40 MINUTES. AS WE  
CHANGED PARTICLE SIZES FOR THE  
P-98 SO DID THE INDEX, IS OUR  
COVERAGE IN THE APPLICATION OK  
OR DO WE NEED TO DO MORE. ALSO  
INCLUDED IS INFORMATION ON THE  
ENCAPSULATED SODIUM CHLORIDE OR  
CROSSLINK LOST CIRCULATION PLUGS.



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ATTACHMENT B

TUC-26 A

# Activity Index Comparison

<b>Magnesium Oxide</b>	<b>Grade</b>	<b>Activity Index, sec</b>	<b>Median Particle Size, Microns</b>
<b>98 HR</b>	<b>Lightburned</b>	<b>9</b>	<b>3</b>
<b>Magchem 30</b>	<b>Lightburned</b>	<b>25</b>	<b>4</b>
<b>P98</b>	<b>Deadburned</b>	<b>840</b>	<b>5</b>
<b>P98</b>	<b>Deadburned</b>	<b>1,410</b>	<b>10</b>
<b>P98</b>	<b>Deadburned</b>	<b>1,740</b>	<b>20</b>
<b>P98</b>	<b>Deadburned</b>	<b>1,890</b>	<b>30</b>
<b>P98</b>	<b>Deadburned</b>	<b>2,940</b>	<b>50</b>
<b>P98</b>	<b>Deadburned</b>	<b>5,610</b>	<b>150</b>